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ESTIMATION OF CONJUGATE γ AND γ' COMPOSITIONS IN Ni-BASE SUPERALLOYS

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ABSTRACT

To control the formation of unwanted phases, superalloy metallurgists have developed methods of estimating the composition of the matrix phase of alloys. That composition is then used to estimate the alloy's propensity toward sigma and other unwanted phase formations upon prolonged exposure to elevated temperatures in service. This paper reviews two approaches for estimating phase composition from the melt composition. One method is based on assigning essentially fixed stoichiometry to precipitating phases and is typified by "PHACOMP." The second method uses analytical geometry to interpret phase diagrams and is shown to be applicable to a two-phase region of a six-component Ni-base system. The geometric method is also shown to be applicable to commercial Ni-base superalloys.

INTRODUCTION

The nickel-base superalloys used in the hot section of gas turbines have been developed to the point where they have useful strength at approximately 80 percent of their melting point (ref. 1). Over 50 such alloys are commercially available.

Typical nickel-base superalloy contain nickel, aluminum, chromium, titanium, zirconium, carbon, and boron. In addition molybdenum, tungsten, niobium, tantalum, hafnium and other reactive or refractory metals may be added to the melt. The two major phases in the alloys are a face centered cubic matrix, γ, and a dispersed, usually coherent, ordered face centered cubic phase, γ'. Small amounts of carbides and borides (usually <3 percent) and other intermetallic compounds are frequently

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present and are responsible for profound changes in mechanical properties. These alloys are clearly described as multicomponent-multiphase alloys typically containing 6 to 13 intentionally added alloy elements and three to five phases.

Since the sigma phase instability was identified in Ni-base superalloys (ref. 2) and the presence of sigma was correlated with a loss of stress rupture life (refs. 3 to 6), the superalloy industry has sought methods to prevent or minimize its occurrence in the alloys. This paper deals with two approaches which have been offered to control the melt compositions to minimize the occurrence of sigma and other undesirable phases in superalloys. The first approach, as exemplified by "PHACOMP" (ref. 7) and the method of reference 8 uses a chemical stoichiometric calculation to estimate the composition of the γ phase. The propensity of the alloy toward phase instability is then determined by calculating the average electron vacancy concentration ($\overline{N}_v$) of the γ. The second approach is to estimate the composition of the conjugate phases (γ and γ') by applying analytical geometry to existing phase diagrams (refs. 9 to 11). The analysis is continued to determine if the melt composition is likely to fall in a phase field containing unwanted phases. In both methods, an attempt is made to estimate the composition of at least one of conjugate phases (usually γ) from the melt composition.

This paper will review the stoichiometric and geometric approaches for estimating the compositions of conjugate phases in superalloys. It will also show that the geometric approach can be used to develop a description of a two-phase region of a six-component system and show that the description of the six-component system can be applied to commercial Ni-base superalloys.

STOICHIOMETRIC APPROACH

Methodology. - The first efforts to control sigma phase in superalloys were those of Boesch and Slaney (ref. 8) and Woodyatt and coworkers (ref. 7). These methods made use of a two step computation. In the first step the melt composition was used to calculate a "residual matrix" or γ-phase composition. In the second, the $\overline{N}_v$ of the γ was compared to an empirically determined value to estimate the propensity of the alloy to form unwanted phases. The procedure is exemplified by the method described in reference 12 which follows:

A. Calculate γ matrix composition in atomic percent:

  1. Calculate composition of carbides and borides as follows:

    a. Half the carbon forms mono-carbides in the sequence TaC, NbC, ZrC, TiC, and VC.

    b. Half the carbon forms either $Cr_{23}C_6$ or $Cr_{21}(Mo, W_2)C_6$ if Mo or W are present. If Mo + W is greater than 6 weight percent, $[NiCo_2(Mo, W)_3]C$ forms instead of $M_{23}C_6$.

    c. A boride, $(Mo_{0.5}Ti_{0.15}Cr_{0.25}Ni_{0.10})_3B_2$ forms.

    d. All of the Al, Ti, and Nb remaining following formation of carbides and borides plus 3 percent of the melt's Cr form γ' of the stoichiometry $Ni_3(Al, Ti, Nb, Cr)$. If there is insufficient Ni, B will also form having a stoichiometry Ni(Al, Ti, Nb, Cr).

    e. The residual γ matrix resulting from subtracting the amount of compounds in (a) to (d) is scaled to 100 percent.

B. The $\overline{N}_v$ of the residual γ matrix is calculated by

$$\overline{N}_v = \sum_{i=1}^{n} f_i (N_v)_i \qquad (1)$$

where

f_i	atomic fraction of the nth element
$(N_v)_i$	4.66 for Cv, Mo, W; 3.66 for Mn; 2.22 for Fe; 1.61 for Co 0.61 for Ni

($(N_v)_i$ may be taken as 10.66 - group number if no value is known.) If N_v is approximately 2.45 or greater the alloy is prone to form σ or other undesirable phases.

Discussion. - This method has been successfully used to control the melt composition of several commercial superalloys (ref. 12). While it would be interesting to review all the additional work in this area, it is considered beyond the scope of this paper and interested readers are referred to references 12 to 17. To summarize the additional work, it should be noted that much of it has been directed at finding a universal critical $\overline{N}_v$, that is, a value of $\overline{N}_v$ appropriate to all alloys or toward finding a systematic method of defining the critical $\overline{N}_v$ for a particular melt. It has been proposed that for some elements' N_v be altered and that the γ' composition be altered from those first proposed. No method has been successful in finding a γ' composition and a series of elements' N_vs which can properly estimate σ and other instabilities at a single value of $\overline{N}_v$ for all alloys.

It should be noted that the methods which rely on a nearly fixed γ' stoichiometry necessarily assume that no Al, Ti, Nb (and perhaps other γ' formers), C and B are soluble in the γ. Mihalisin and Pasquine (ref. 17) suggested that this treatment of γ' was in error and could account for problems observed in predicting the stabilities of alloy 713C and alloy 713LC. They suggested that actual chemical analysis of the γ' phase be made and that a mass balance, also using the measured volume fraction of γ', be used to calculate the residual γ matrix composition. The propensity toward forming σ phase of that γ was estimated in a manner similar to that described earlier for PHACOMP in that the $\overline{N}_v$ was calculated and compared to an empirically selected value. The presence of significant Al in the γ was also noted in reference 18 for Udimet 700. While the method of reference 17 is likely to better estimate the γ composition than PHACOMP, it suffers from the need for preparing a melt from which the γ' can be analyzed. Therefore the method of reference 17 is of reduced value in alloy development programs.

GEOMETRIC APPROACH

The Ni-Al-Ti-Cr System at 750° C

Background. - The work of Kriege and Baris (ref. 19) confirmed the presence of significant Al and Ti in the γ phase of 15 commercial Ni-base superalloys. Further, their work showed, while γ' was nearly stoichiometric, that variations in its compositions occur which could affect a PHACOMP calculation.

In an effort to avoid reliance on a fixed or predictable γ' composition to predict γ composition, references 9 to 11 used analytic geometry to interpret the Ni-Al-Ti-Cr phase diagrams of Taylor and co-workers (refs. 20 to 23). The problem addressed that of finding the composition of the γ phase, given the melt composition and assuming the melt lies in the two-phase ($\gamma + \gamma'$) field. The approach was to find the intersection of the tie line on which the two-phase alloy lies and the γ - γ' solvus surface (the locus of γ compositions saturated with respect to γ').

Methodology. - As a first step the γ - γ' solvus surface of the Ni-Al-Ti at 750° C was curve fit using the method of least squares. This equation was then altered to fit the Ni-Al-Ti-Cr γ - γ' solvus resulting in the equation

$$\mathrm{Al} + 1.5\mathrm{Ti} = 1.33\mathrm{Cr}^2 - 0.566\mathrm{Cr} + 0.12 \qquad (2)$$

That equation (2) is a good simulation of the γ - γ' solvus at low Ti concentrations can be seen in figure 1 by comparing the solid lines surface (eq. (2)) and the double-dashed line from reference 23.

The next step was to deduce the tie line behavior in the alloy systems. Fortunately, Taylor and Floyd showed some tie lines in their ternary diagrams in references 20 to 22. The tie line system in the four component systems has to include the tie lines of the three component systems as a limit and further the tie lines in the four component systems must not intersect each other in the two-phase field. The details of the system proposed are described in references 10 and 11. The procedure will now be briefly reviewed for the Ni-Al-Ti-Cr system at 750° C. Three assumptions are made to allow a closed form solution:

1. Assume that for the system Ni-Cr-Al tie lines (for $\gamma + \gamma'$) intersect at (Ni, 0.25Al) (fig. 2).

2. Assume that for the system Ni-Cr-Ti that η and γ' are mutually soluble and tie lines intersect at (Ni, 0.25Ti) (fig. 3).

3. Assume that for the system Ni-Al-Ti that $\gamma + \gamma'$ tie lines intersect at (Ni, -0.05Al) (fig. 4). (Also shown as point S in fig. 1.)

In the specific construction to be used, a tie line is formed by the intersection of two planes. One plane has a zone axis (for this paper defined as a line common to all planes in the set) from the Cr corner to point S in figure 1. The other plane has line U-V as a zone axis (fig. 1). The Al to Ti to Ni ratio of the alloy governs the selection of the plane from the Cr-S set and the Cr concentration selects the plane from the U-V set.

Because U-V and Cr-S are zone axes for the two planes determining the tie line, the only places where the tie line families can intersect are on U-V or Cr-S. Both of these lines are outside of the two-phase field; therefore, Gibbs' phase rule is followed.

The details of the construction by which the composition of the γ phase in a two-phase quaternary alloy is determined from the above analysis follows. In figure 1 the composition of the two-phase quaternary alloy is projected on the Ni-Al-Ti diagram by passing a line from the Cr corner of the quaternary, through the two-phase alloy A to the Ni-Al-Ti ternary. This construction locates a point T in the ternary diagram that has the same relative Ni, Al, and Ti concentration as in the quaternary alloy. This is shown as line Cr-A-T. The intersection of U-V (the line connecting 0.25Al, 0.75Ni, and 0.25Ti, 0.75Ni) and T-S (the tie line in the Ni-Al-Ti ternary) established point X on the tie line (containing the two-phase quaternary alloy A). The tie line on which alloy A lies is X-A-G. The composition of G (γ phase) is determined by finding the intersection of X-A with the solvus surface.

The mathematics of this treatment can be followed in reference 10 or 11. The approach was adapted to commercial superalloys by using a stoichiometric approach to account for carbides, borides, and γ' formed by Nb, Ta, Zr. and V. The Mo and W in γ were treated by analogy to Cr. Comparison of γ predicted by the geometric method for the alloys in Table I and that determined by references 18 and 19 are shown in Table II. Note that both Co and Ti were in error by a constant amount and factor, respectively.

Discussion. - While the application of the procedure to commercial alloys was encouraging, it required some extra assumptions. It was felt that the general techniques of using analytic geometry to interpret two-phase regions of multicomponent phase diagrams had been demonstrated.

The Ni-Al-Cr-Ti-W-Mo System at 850° C

Background. - Analysis of the work of references 10 and 11 suggested that a better superalloy phase diagram could be developed by including W and Mo directly in the system's phase diagram. Further, it was clear from references 10 and 11 that to predict conjugate phase compositions it was not necessary to graph a phase diagram, only to describe its significant features by mathematical expressions. These ideas from references 10 and 11 coupled with a quantitative analytical procedure for γ' (ref. 19) encouraged the author to initiate a study to determine the compositional limits of γ and γ' in the Ni-Al-Cr-Ti-W-Mo system at 850° C (refs. 24 and 25). The temperature of 850° C was selected as it was thought to be near the temperature at which σ and related phases precipitate rapidly (refs. 4 to 6).

Experimental approach. - The experimental details of the study are available in reference 25. They will only be summarized here. After checking two preliminary alloys, a series of melts were made, the compositions of which were based on a fraction of a three-level factorial design (ref. 26). The experiment design is shown in Table III. The compositions of the two-phase alloys melted are shown in Table IV. The alloys were homogenized for 4 hours at 1190° C and air cooled to room temperature. They were heated at 850° C for 1008 hours before being air cooled to room temperature. The γ' was quantitatively extracted from the heat treated two-phase alloys using the method of reference 19. After the γ' residues and the alloys were chemically analyzed, the composition of γ was calculated by a mass balance.

This experimental approach allowed the determination of the conjugate γ and γ' compositions with a single melt. The solvus hypersurfaces were then described by fitting a third degree equation to the γ and γ' compositions using linear regression analysis (ref. 27).

The average composition and amount of γ' are shown in Table V. The range of composition is consistent with that of other investigations (refs. 17 to 19, and 28 to 30). Also shown in Table V is the sample standard deviation (S) for each element except Ni. The value of S is less than one-third the range of each element observed which suggests the spread in observed compositions is significant when compared to analytical errors.

The average composition of γ is shown in Table VI. The sample standard deviation (S), also shown in Table VI for each element can be seen to be less than one-third the range of each element, again suggesting that the observed spread is greater than would be expected from experimental errors. The range of γ compositions is consistent with the ranges observed in other work (refs. 17 to 19, and 28 to 30).

Gamma-gamma prime relationship. - The compositions of the γ in Table VI and γ' shown in Table V are compositions of the phases from two-phase alloys. These compositions, therefore, represent points on the solvus hypersurfaces, and when considered in pairs (one γ and one γ') from a heat, these are, in fact, the compositions of conjugate phases.

To obtain a more useful description of these solvus hypersurfaces, the data points from each extraction were fitted with curves using a multiple linear regression computer program (ref. 27). The model equation used to fit both sets of data was:

$$
\begin{aligned}
\mathrm{Al} = {} & B_0 + B_1 \times \mathrm{Cr} + B_2 \times \mathrm{Mo} + B_3 \times \mathrm{Ti} + B_4 \times \mathrm{W} \\
& + B_5 \times \mathrm{Cr}^2 + B_6 \times \mathrm{Mo}^2 + B_7 \times \mathrm{Ti}^2 + B_8 \times \mathrm{W}^2 \\
& + B_9 \times \mathrm{Cr} \times \mathrm{Mo} + B_{10} \times \mathrm{Cr} \times \mathrm{Ti} + B_{11} \times \mathrm{Cr} \\
& \times \mathrm{W} + B_{12} \times \mathrm{Mo} \times \mathrm{Ti} + B_{13} \times \mathrm{Mo} \times \mathrm{W} + B_{14} \\
& \times \mathrm{Ti} \times \mathrm{W} + B_{15} \times \mathrm{Cr} \times \mathrm{Mo} \times \mathrm{Ti} + B_{16} \times \mathrm{Cr} \\
& \times \mathrm{Mo} \times \mathrm{W} + B_{17} \times \mathrm{Cr} \times \mathrm{Ti} \times \mathrm{W} + B_{18} \times \mathrm{Mo} \\
& \times \mathrm{Ti} \times \mathrm{W} + \mathrm{error} \qquad (3)
\end{aligned}
$$

where Al, Cr, Mo, Ti, and W are in atomic percent, B_0 is a constant, and B_1, B_2, . . ., B_{18} are coefficients.

For both solvus hypersurfaces, the regression program rejected coefficients with less than a 25-percent significance level. The low significance level was chosen because it is recognized that the independent variables have error associated with them. Although it is desirable to simplify the equations, the regression analysis assumption that the independent variables are known without error is violated. The low significance level is believed to avoid rejecting significant terms.

The constant and coefficients for both solvus equations are shown in Table VII. The multiple regression coefficient (R^2) for the γ is 0.89 and for the γ', R^2 is 0.87.

These two equations may be used to plot sections of the hypersurfaces or simply to estimate the amount of a particular element in a phase if four others are known. The usefulness of these equations could be increased if they could be used to estimate the compositions of conjugate phases, given the composition of a two-phase alloy.

The compositions obtained in this investigation for γ and γ' are the compositions of conjugate phases. Therefore a tie line is known to pass through the γ composition, the alloy composition and the γ' composition. Direction numbers for the tie lines were calculated by:

$$DN_i = \frac{I_\gamma - I_{\gamma'}}{Cr_\gamma - Cr_{\gamma'}} \qquad (4)$$

where

DN_i	direction number for the ith element
I_γ	composition of I in γ
$I_{\gamma'}$	composition of I in γ'
Cr_γ	composition of Cr in γ
$Cr_{\gamma'}$	composition of Cr in γ'

These direction numbers for each element (Cr being 1) indicate the change in amount of the element along a tie line per unit change in Cr. These, in effect, describe the slope of the tie lines.

To determine the phase compositions from the alloy composition by using the tie lines, the direction number of the tie line needs to be known as a function of the composition of the alloy. The direction numbers were estimated from the alloy compositions by using a multiple linear regression program (ref. 27) to fit the direction numbers for Al, Mo, Ti, and W to equations of the form:

$$DN_i = B_0 + B_1 \times Al + B_2 \times Cr + B_3 \times Mo + B_4 \times Ti + B_5 \times W + \text{error} \qquad (5)$$

The full model was used since the uncertainties involved in rejecting terms of low significance seemed large when compared to the small gain obtained in simplifying these equations. The values of the constants and coefficients for these equations are summarized in Table VIII.

The two-phase region of this alloy system can now be described by using the equations for the solvus hypersurfaces and those which relate the direction numbers of the tie lines to the alloy chemistry. In principle, these equations could be solved simultaneously to find the composition of the γ and γ' for an alloy of known composition. This approach was not used because errors resulting from the least squares curve fitting were expected (and did) result in conditions where the tie lines fail to intersect the solvus hypersurfaces. Furthermore, because the solvuses are parabolic in shape, it is possible that two real and positive solutions exist.

The procedure used to find the compositions of the conjugate phases from the composition of a two-phase alloy is described below. It was programmed in FORTRAN IV for a time-sharing IBM 360 computer (ref. 25). First, the composition of the alloy is used to establish direction numbers for Al, Mo, Ti, and W by using the equations from Table VIII. Next, the alloy composition is changed by an increment of Cr and the new values for the other elements are calculated from the direction numbers. The composition is therefore still on the tie line. The new values of Cr, Mo, Ti, and W are used in the solvus equation to calculate the Al for the solvus, if the other four elements were as just estimated. This procedure is repeated until the Al compositions on the tie line and on the solvus agree to within 0.005 percent or until it is obvious that no intersection will be found. If it appears that a second solution is likely, the procedure is repeated. If no intersection is located, the closest approach of the tie line to the solvus (as defined by the least difference in Al) is displayed as a solution.

To solve for γ composition, the Cr is increased from the alloy composition. To solve for the γ' composition, the Cr is decreased from the alloy composition. The closest approach is taken as a solution if no intersection is found for γ when Al is 0 percent or Cr is 40 percent. The closest approach is used for γ' if Al is 30 percent or Cr is 0 percent. Where two intersections were found, the higher Cr solution for γ and the lower Cr solution for γ' appeared to be closer to the experimental values.

The results of this calculation for the two-phase experimental alloys are compared to the experimental results in Table IX. The experimental compositions and those calculated by the phase analysis procedure are in good agreement. Except for γ composition in two alloys (ref. 25) the agreement was excellent. It is assumed that the two exceptions were the result of accumulated error.

Occurrence of additional phases. - The primary purpose of the investigation reported in references 24 and 25 was to define the $\gamma + \gamma'$ region of the Ni-Al-Cr-Ti-W-Mo system. During the conduct of the investigation, 51 alloys were melted. Two alloys were determined to contain only γ, 27 alloys contained only γ and γ' and the remaining 22 alloys were multiphase alloys containing phases other than γ and γ'. This section will relate primarily to the latter group of multiphase alloys which contained from three to six identifiable phases.

The composition of the multiphase alloys and phases (other than γ and γ') present are listed in Table X. The phases observed were σ, μ, and two body centered cubic phases, one having a lattice constant (A_0) similar to Cr, the other having an A_0 similar to W and Mo. No alloy in Table X contains more than 74.5 percent Ni and only alloys 3 and 44 contain in excess of 70 percent Ni. When one converts the two-phase alloys of Table IV to atomic percent, it is seen that these alloys had greater than 65.5 percent Ni and all but two alloys had greater than 67 percent Ni. This compares well with the γ - σ boundary of 62 to

64 percent Ni + Co reported in reference 31 and the $\gamma - \alpha$ boundary of 60 percent in reference 20, see figure 5.

While the alloy composition offers a quick check for stability it should be more revealing to examine the γ composition as in PHACOMP. Instead of using an N_V approach, however, a more direct treatment of the phase diagram is proposed here. Reference 31 shows that Mo and W are 1.75 more potent than Cr in promoting σ formation. By using an analogy to the 67 percent Ni limit for two-phase alloys, it is suggested that the [Cr + 1.75(Mo + W)] for the γ should be less than 33 percent (100 - 67 percent). For the stable alloys in Table VI, only two (alloy 15 and alloy 37) have values of the function Cr + 1.75(Mo + W) which were greater than 33 percent. The maximum value was 36.1 for alloy 15. For the multiphase alloys, the γ composition could only be estimated by use of the computer program previously mentioned from reference 25. The lowest value of the function was 20.2 percent but only three alloys of 14 for which a computation could be made, had values less than 30 percent.

A third parameter which appears capable of assisting in the prediction of additional phases is the rate of change of Al with respect to Cr (Al direction number) along the $\gamma + \gamma'$ tie line. For the two-phase alloys, only two alloys had Al direction numbers which measured greater than -0.30, the maximum value being -0.22. For the multiphase alloys, the minimum value estimated from the computer program of reference 25 was -0.20. Eight of 22 alloys had values greater than 0.0.

The relationship of the direction number of a tie line to a phase boundary is shown in figure 5. The line Q-R is the boundary between a two-phase $(\gamma + \gamma')$ field and a three-phase $(\gamma + \gamma' + \alpha)$ field. It can be seen in the $\gamma + \gamma'$ field as Cr increases from the Ni-Al binary to line Q-R, the Al direction number increases from negative ∞ to -0.74. While the difference between the experimental limit of about -0.30 does not compare too well with that predicted by the Ni-Al-Cr phase diagram, one can see that there is a basis for the concept.

The three parameters for estimating occurrence of multiphase alloys are shown in figure 6. They are shown schematically as a Venn diagram to imply that as more than one of the conditions is satisfied, the probability of occurrence of additional phases increases.

APPLICATION TO COMMERCIAL ALLOYS

Phase diagram. - The alloys studied in reference 19 are typical of current commercial Ni-base superalloys. The heat treatments for these alloys, except for Udimet 700, are typical of the condition in which the alloys may be placed in service. In addition to the six elements studied in the last investigation, the commercial alloys in reference 19 contain C and may have intentional additions of Co, Nb, Fe, Ta, and V. The compositions from reference 19 were used to determine whether the "phase diagram" could be applied to commercial alloys.

The compositions from reference 19 were first converted to atomic percent to test the "phase diagram." The composition was then adjusted for carbide formation by using the procedures suggested in reference 32. The adjusted composition was then treated as an alloy composition using the computer program described earlier. The procedure in effect treated all elements other than Al, Cr, Mo, Ti, and W as if they were Ni. This appears to be a reasonable assumption for Co and Fe, but Ta and Nb are shown in reference 19 to be γ' formers.

The results of these calculations of γ and γ' compositions are compared to the compositions reported in reference 19 in Table XI. The compositions of γ calculated compared well with those reported except for alloys IN 100, Mar M200, Nimonic 115 and Nicrotung. For the γ' compositions, only alloys Inconel X-750 and Unitemp AF 1753 failed to show good agreement between the calculated and observed values. For alloy Unitemp AF 1753, the estimating procedure reported the alloy composition for the γ composition. This can be considered to indicate the alloy to be single phase.

The "phase diagram" of this investigation is capable of describing one phase in all of the commercial alloys examined. Of the six phase analyses which were not in reasonable agreement, four were for the γ phase. This is probably because the γ' composition was directly determined in both this investigation and reference 19 and a greater uncertainty should exist for the composition of the γ phase. The two alloys for which the γ' estimate was poor had the lowest weight fraction γ' of the alloys examined. Errors in estimating the tie line direction numbers would be expected to be magnified in the composition of the phase more distant from the alloy composition because of a leverage effect.

The results of the above comparisons indicate that the techniques developed in this investigation should be capable of being adapted for use in commercial alloys. It appears that the discrepancies between the estimates based on the current work and reference 19 are partly the result of the fact that the alloys in reference 19 were heat treated for shorter times and at different temperatures than the current work. The other obvious source differences is that the current work made no attempt to account for additional elements, except as they enter into carbide reactions.

Additional phases. - The alloys of reference 19 were also used to test the applicability of the parameters related to formation of additional phases. The three parameters (Ni in alloy; Cr + 1.75(Mo + W) in γ; and Al direction number on the $\gamma + \gamma'$ tie line) were calculated using the data from reference 19. These parameters are shown in Table XII. Also shown in Table XII are the phases that Collins and Kortovich (ref. 15) observed in these alloys. (It should be noted that the composition of the alloys varied slightly between refs. 15 and 19.) Reference 19 did not report on the stability of the compositions.

While the phase boundary between the two-phase and multiphase alloys is not identical to that of reference 25, it can be seen that the three parameters show similar critical regions. Alloys with less than 67 percent Ni+Co+Fe were multiphase alloys. γ phase with Cr + 1.75(Mo + W) greater than 31.5 formed additional phases. While it is difficult to find a critical value for the Al direction number, the average value for alloys having a high propensity toward forming σ and μ is -0.44 while it is -0.55 for the stable alloys. The difference between the averages is significant at the 95 percent level.

The three parameters identified in reference 25 do appear to be appropriate in determining if commercial alloys are likely to form undesirable phases. The specific composition of IN 100, which is shown from reference 15 as a σ former in Table XII, may have been stable in reference 19. IN 100 is one of the alloys which may be stable or σ forming within its normal composition limits (refs. 3 to 6).

CONCLUDING REMARKS

A review of two methods of estimating conjugate phase compositions in Ni-base superalloys has been presented. Both methods had as an ultimate objective the estimation of a melt's propensity for forming unwanted phases such as σ.

One method as exemplified by PHACOMP uses the chemical stoichiometric approach. This method reduces from the melt composition the elements that are believed to precipitate and arrives at the residual matrix by difference. The stability of the alloy is determined by calculating the average electron vacancy concentration ($\overline{N}_v$) of the residual matrix and comparing that with an empirically determined critical $\overline{N}_v$.

The method has been successfully used as a quality control tool for several commercially produced alloys where the range of compositions is limited and the critical $\overline{N}_v$ was empirically determined. However, when used as a research tool, no single γ' composition or elemental $\overline{N}_v$'s could be found to allow one critical $\overline{N}_v$ for sigma formation. One reason for this problem is that γ' has been experimentally shown to have a variable stoichiometry and perhaps of greater significance is that γ' formers are soluble in γ in varying amounts.

It is believed that the stoichiometric approach also can be successfully used in systems where the precipitate is in essence a line compound (solvus independent of composition), has little solubility in the second phase, or is present in small amounts. This may be the case for the carbides and borides in the Ni-base superalloys.

The analytical geometric approach to estimating the composition of conjugate phases has been demonstrated for Ni-base alloys containing up to six components. The work reviewed here has shown that the two-phase region ($\gamma + \gamma'$) of the Ni-Al-Cr-Ti-W-Mo systems at 850° C can be satisfactorily modeled. If one is given a melt composition the composition of the conjugate γ and γ' can be determined from the model. Furthermore, I believe that the approach that was applied to a six-component Ni-base system can be used in either higher order Ni-base systems and in other alloy bases.

In developing the geometric approach for estimation of conjugate phase compositions, it was observed that the propensity toward formation of additional phases could be estimated from parameters available from the geometric analysis. Specifically it was observed that the Ni in the alloy, the parameter Cr + 1.75(Mo + W) and the Al direction number along the $\gamma + \gamma'$ tie line, can be used to estimate the propensity of an alloy toward additional phase formation. All three parameters can be related to solvus curves on conventional phase diagrams. It is suggested that additional study in relating phase diagram parameters to solvus surfaces can further refine this approach and make it applicable to different alloy bases, higher order systems and perhaps three or four phase regions. For years the phase diagram literature has touched on mathematical modeling of systems, however, in practice the industry has settled for what it can draw on a plane sheet or model in three-dimensional space.

In closing, I would like to offer that the work reviewed here should encourage more work on multicomponent systems where the computer can allow interpretation of the modeled diagrams. This approach has been demonstrated here and its extension to commercial alloys has been indicated.

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TABLE I. - COMPOSITION OF ALLOYS

Alloy	Reference	Heat treatment	Weight percent (balance is nickel)											
			Cr	Co	Al	Ti	W	Mo	Nb	Ta	V	Fe	C	B
B-1900	19	1	7.9	9.8	5.9	1.0	----	5.7	-----	4.5	----	----	0.09	----
GMR 235		1	15.9	-----	3.5	2.0	----	5.0	-----	---	----	9.8	.15	----
Inconel 700		2	14.3	28.5	3.0	2.5	----	3.9	-----	---	----	.7	.12	----
Alloy 713C		1	12.6	-----	6.8[a]	.8	----	4.7	2.1	---	----	----	.16	----
Inconel X-750		3	14.6	-----	.8	2.4	----	----	.8	---	----	6.5	.04	----
IN 100		1	9.8	15.0	5.6	5.7[a]	----	3.1	-----	---	0.9	----	.19	----
Mar-M 200		1	8.9	9.5	4.5	1.9	12.3	----	1.1	---	----	----	.16	----
Nicrotung		1	11.0	9.9	4.4	4.2	8.0	----	-----	---	----	----	.07	----
Nimonic 115		4	14.8	14.8	4.8	3.9	----	3.5	-----	---	----	----	.14	----
René 41		5	19.0	10.7	1.5	3.1	----	9.7	-----	---	----	----	.09	----
TRW 1900		1	10.1	10.3	6.7	1.0	9.2	----	1.6	---	----	----	.14	----
Udimet 500		6	18.7	19.3	2.9	3.0	----	4.3	-----	---	----	----	.07	----
Udimet 700		7	15.4	18.8	4.4	3.4	----	5.0	-----	---	----	----	.06	----
Unitemp AF 1753		8	16.4	7.7	2.0	3.4	8.3	1.5	-----	---	----	9.0	.23	----
Waspaloy		9	18.6	13.0	1.4	2.9	----	4.2	-----	---	----	----	.05	----
Alloy 713C	17	1	13.23	-----	5.86	.79	----	4.46	[b]2.09	---	----	.10	.11	0.01
Alloy 713LC (heat 07)		1	12.32	-----	5.90	.72	----	4.46	[a]2.13	---	----	.20	.06	.01
Alloy 713LC (heat 17)		1	12.52	-----	5.90	.60	----	4.41	[a]2.10	---	----	.15	.03	.01
IN 731 X		1	9.60	9.72	5.60	4.66	----	2.46	-----	---	.85	.23	.16	.01

Heat treatment	Description
1	As cast
2	2160° F/ 2 hr/air cool + 1600° F/ 4 hr/air cool
3	2100° F/ 2 hr/air cool + 1550° F/ 24 hr/air cool + 1300° F/20 hr/air cool
4	2175° F/$1\frac{1}{2}$ hr/air cool + 2010° F/ 6 hr/air cool
5	1950° F/ 4 hr/air cool + 1400° F/ 16 hr/air cool
6	1975° F/ 4 hr/air cool + 1550° F/ 24 hr/air cool + 1400° F/16 hr/air cool
7	2140° F/ 4 hr/air cool + 1800° F/136 hr/air cool
8	2150° F/ 4 hr/air cool + 1650° F/ 6 hr/air cool
9	1975° F/ 4 hr/air cool + 1550° F/ 24 hr/air cool + 1400° F/16 hr/air cool

[a] Value higher than AMS specification for alloy.

[b] Reported in ref. 17 as Nb + Ta.

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TABLE II. - COMPARISON OF PROPOSED METHOD AND ANALYZED DATA

Element	Average difference, $\bar{d}$,[a] wt %	Sample standard deviation of difference, s_d
Cr	1.1	2.05
Al	.007	.57
Ti[b]	.007	.28
Co[b]	.017	2.74
Mo	.136	1.36
Fe	1.38	1.10
W	-3.73	3.52

[a] $\bar{d} = \sum_{i=1}^{n} \frac{\text{literature - calculated}}{n}$.

[b] $\bar{d}$ was forced to be essentially zero.

TABLE III. - EXPERIMENTAL DESIGN

Element	Level	Alloying addition, at.%, Ni is balance, heat number	
		1-36	37-49
Al	Low	4.0	4.0
	Medium	9.0	8.0
	High	13.0	12.0
Ti	Low	0.25	0.25
	Medium	1.75	1.75
	High	4.75	3.75
Cr	Low	6.5	6.5
	Medium	13.5	12.5
	High	20.5	18.5
W	Low	0	0
	Medium	2.0	1.5
	High	4.0	3.0
Mo	Low	0	0
	Medium	3.0	2.0
	High	6.0	4.0

TABLE IV. - COMPOSITION OF

TWO-PHASE ALLOYS

Heat	Composition, wt %					
	Al	Cr	Mo	Ni	Ti	W
98	2.9	9.4	0	76.9	1.5	9.3
99	3.9	11.2	9.3	73.9	1.7	0
2	4.1	12.5	4.8	78.4	.2	0
5	1.5	5.9	4.2	75.7	1.3	11.2
7	3.9	6.8	0	80.0	3.3	6.0
12	3.7	5.5	4.6	72.4	.1	13.7
14	6.4	5.7	0	81.0	1.4	5.5
15	2.2	20.0	5.1	65.1	1.5	6.1
16	1.9	6.5	10.2	77.4	3.9	0
[a]21	1.7	6.2	0	85.9	.2	5.9
22	1.8	12.1	5.2	79.4	1.5	0
23	4.8	6.3	10.8	76.6	1.5	0
28	7.4	6.3	0	74.3	.2	11.9
31	4.7	17.7	0	70.6	1.2	5.8
33	1.9	5.9	9.6	75.7	1.2	5.7
34	2.1	13.3	0	80.7	3.9	0
35	4.7	5.4	4.8	81.4	3.6	0
37	3.9	13.2	6.8	70.2	1.3	4.6
38	6.5	12.8	4.0	73.5	3.2	0
39	5.4	6.6	6.8	81.1	.1	0
40	5.9	11.2	0	72.6	1.6	9.0
41	1.8	12.3	6.7	72.2	3.0	4.0
42	4.1	18.9	3.7	68.9	.1	4.3
46	4.1	10.9	7.9	68.6	.3	8.2
47	5.6	11.5	3.5	73.2	1.3	4.8
48	3.6	17.5	0	75.2	3.8	0
49	5.5	17.2	0	75.6	1.7	0

[a] Alloy contain less than 1 percent γ' - no analysis of γ' could be made.

TABLE V. - COMPOSITION AND AMOUNT OF GAMMA PRIME

Heat	Element, at.%						Amount of γ', wt%
	Ni	Al	Cr	Mo	Ti	W	
98	78.0	12.5	2.7	---	3.7	3.1	40.1
99	76.1	13.8	2.6	2.9	3.9	---	39.4
2	74.5	17.3	5.1	2.4	.7	---	29.3
5	75.7	13.3	2.1	1.1	5.1	2.7	[a]5.6
7	76.9	11.7	2.4	---	7.1	1.9	39.2
12	77.8	14.6	1.9	1.5	.3	3.9	31.9
14	73.3	15.9	4.2	---	2.8	3.8	18.4
15	74.3	11.3	4.9	1.1	6.3	2.1	16.8
16	75.6	10.3	1.5	2.3	10.3	---	20.3
22	76.2	7.8	8.9	2.2	4.9	---	8.5
23	74.2	15.9	2.7	3.9	3.3	---	44.5
28	72.1	16.9	3.5	---	.3	7.2	32.1
31	74.3	16.0	4.0	---	3.3	2.4	41.3
33	77.1	10.8	2.4	2.4	5.9	1.4	[a]1.4
34	73.1	10.1	2.9	---	13.9	---	21.5
35	77.3	12.1	2.3	1.7	6.6	---	54.5
37	74.7	15.7	2.7	1.7	3.5	1.7	35.3
38	74.7	15.1	3.5	1.6	5.1	---	34.2
39	77.5	15.5	3.2	3.5	.3	---	44.7
40	76.0	15.1	3.3	---	2.3	3.3	55.7
41	72.4	10.6	2.1	2.9	9.5	2.5	23.9
42	73.6	17.5	4.6	1.3	.3	2.7	27.1
46	76.6	14.9	2.9	1.7	.4	3.5	29.3
47	75.8	15.3	3.2	1.3	2.4	2.0	55.6
48	76.1	13.1	3.9	---	6.9	---	38.7
49	75.9	15.3	2.7	---	2.7	---	51.9
S		0.851	0.336	0.573	0.502	0.852	

[a] Electrolyte was H_3PO_4 - extraction not quantitative in amount of γ'.

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TABLE VI. - COMPOSITION OF GAMMA

Heat	Element, at.%, balance is Ni				
	Al	Cr	Mo	Ti	W
98	2.1	16.4	---	0.5	2.9
99	4.3	19.1	7.5	.8	---
2	4.7	17.2	3.0	0	---
7	6.1	10.9	---	1.7	1.9
12	5.5	8.9	3.7	.1	5.0
14	12.7	6.6	---	1.5	1.2
15	3.5	26.5	3.6	.9	1.9
16	2.1	9.2	7.5	3.1	---
22	3.4	13.9	3.2	1.5	---
23	5.1	10.7	8.7	.5	---
28	15.4	8.5	---	.1	2.2
31	5.5	29.9	---	.1	1.3
34	2.5	17.7	---	1.9	---
35	6.9	10.3	4.2	1.2	---
37	4.3	21.8	5.5	.5	1.3
38	11.9	18.5	2.7	2.9	---
39	7.6	10.5	4.3	0	---
40	9.5	23.3	---	1.5	2.1
41	1.9	17.8	4.5	1.9	.9
42	5.5	25.7	2.5	.1	.8
46	6.7	16.7	6.3	.4	2.3
47	7.5	24.4	3.1	.5	.8
48	3.5	27.7	---	2.5	---
49	6.2	30.7	---	1.1	---
S	0.640	0.391	0.224	0.256	0.374

TABLE VII. - REGRESSION ANALYSIS OF GAMMA AND GAMMA PRIME

$$\left[\text{Equation: } \mathrm{Al} = \sum_{n=0}^{n=18} B_n \times F_n.\right]$$

n	Phase		Factor, F_n
	γ Coefficient, B_n	γ' Coefficient, B_n	
0	13.3992	7.42647	Constant
1	-1.07392	3.59713	Cr
2	1.80069	0	Mo
3	0	.849058	Ti
4	15.3168	-.589230	W
5	.0318507	-.292157	Cr^2
6	.0455815	.149930	Mo^2
7	1.53473	-.0256415	Ti^2
8	-2.59870	-.0398181	W^2
9	-.100793	-.127831	Cr × Mo
10	-.204796	-.310730	Cr × Ti
11	-.504191	.290021	Cr × W
12	-2.12721	-.245979	Mo × Ti
13	.598921	.876515	Mo × W
14	-7.75600	-.155343	Ti × W
15	.153165	.0675603	Cr × Mo × Ti
16	0	-.275617	Cr × Mo × W
17	.486625	0	Cr × Ti × W
18	-1.43936	.0226054	Mo × Ti × W
R^2	.89	.87	

TABLE VIII. - LEAST SQUARES ANALYSIS OF DIRECTION NUMBERS

$$\left[\text{Equation: Direction number} = \sum_{n=0}^{n=5} B_n \times F_n.\right]$$

n	Element				Factor, F_n
	Al coefficient, B_n	Mo coefficient, B_n	Ti coefficient, B_n	W coefficient, B_n	
0	-2.4316	-0.0032572	-1.04691	-0.52841	Constant
1	.066096	-.0010339	.046595	-.054240	Al
2	.059819	-.013136	.030941	.030041	Cr
3	.0088685	.087981	.026712	.020858	Mo
4	.14622	.021598	-.070565	.054511	Ti
5	.090661	.024404	.040548	.14031	W
R^2	.64	.72	.76	.37	
${}^{a}T$	.35	.09	.96	.29	

[a] T is the significance level of the least significant coefficient.

TABLE IX. - COMPARISON OF ANALYZED DATA AND CALCULATION FOR EXPERIMENTAL ALLOYS

Element	Phase	Average difference, $\overline{d}$,[a] at.%	Sample standard deviation of difference, Sd
Al	γ	-0.50	1.6
	γ'	.59	1.5
Cr	γ	.93	4.8
	γ'	.90	1.8
Mo	γ	.18	.54
	γ'	-.09	.72
Ti	γ	.12	.82
	γ'	.12	1.28
W	γ	.23	.95
	γ'	-.66	1.6

[a] $\overline{d} = \sum_{i=1}^{i=n} \left(\frac{\text{observed - calculated}}{n}\right).$

TABLE X. - OCCURRENCE OF PHASES

Alloy	Element, at.%						Phase			
	Al	Cr	Mo	Ti	W	Ni	Cr	[a]Mo, W	σ	μ
3	14.6	8.4	5.5	0.2	---	71.2				×
4	12.9	11.9	---	1.3	4.1	69.8		×		
6	7.3	21.6	5.6	1.4	3.6	60.5		×	×	
8	13.1	21.9	2.8	4.3	1.8	56.1	×	×	×	
9	4.0	14.8	5.4	5.1	2.1	68.6		×		×
10	9.7	23.5	6.4	.4	4.6	55.4		×	×	
13	8.8	15.2	5.5	1.6	1.8	67.1				×
17	8.6	23.4	---	4.3	---	63.7	×			
18	15.3	14.0	3.0	4.9	---	62.9	×		×	
19	9.9	17.8	3.1	.1	2.1	67.0		×		×
20	15.7	13.1	5.4	.2	2.0	63.5		×	×	
24	14.2	20.7	---	1.4	---	63.6	×			
25	13.3	7.1	2.9	4.6	3.4	68.6		×	×	
26	3.9	21.8	5.6	4.5	3.6	60.6		×	×	×
27	7.1	17.0	---	4.6	4.0	67.2	×	×		
29	4.1	22.7	3.2	.1	3.8	66.1		×		
30	9.8	14.8	5.8	.1	3.8	65.7		×		×
32	14.7	13.7	3.1	1.7	1.9	65.3	×	×	×	
36	14.8	21.0	6.4	4.5	---	55.3	×	×	×	
43	12.9	12.5	4.1	4.9	1.3	64.2				×
44	10.9	6.4	2.0	3.3	2.9	74.5	Unidentified			
45	3.6	19.6	4.1	3.9	2.6	66.2		×		

[a] Mo and W cannot be differentiated by X-ray diffraction. BCC phase with A_0 at approximately 3.15 Å.

TABLE XI. - COMPARISON OF OBSERVED AND ESTIMATED PHASE COMPOSITIONS IN COMMERCIAL ALLOYS

Alloy	Phase	Element, at.%									
		Al		Cr		Mo		Ti		W	
		Obs[a]	Est[b]	Obs	Est	Obs	Est	Obs	Est	Obs	Est
B-1900	γ	5.1	1.8	18.3	20.5	5.4	5.7	0	0	---	---
	γ'	17.2	16.4	3.0	4.4	2.3	2.6	1.9	1.7	---	---
GMR 235	γ	3.8	3.5	20.6	22.5	3.2	3.3	.6	.4	---	---
	γ'	17.6	14.3	2.3	6.0	1.4	2.2	5.1	4.4	---	---
Inconel 700	γ	4.0	4.8	19.4	16.9	2.4	2.3	1.0	1.7	---	---
	γ'	13.6	13.0	4.3	5.7	1.2	1.8	6.7	6.4	---	---
Inconel 713C	γ	8.1	5.4	24.3	27.2	3.9	3.6	.1	.6	---	---
	γ'	19.2	18.0	3.5	6.5	1.5	2.3	1.3	.6	---	---
Inconel X-750	γ	.6	.5	17.9	16.3	---	---	1.2	2.0	---	---
	γ'	6.9	15.5	2.3	1.5	---	---	12.8	12.5	---	---
IN 100	γ	4.8	10.4	24.0	14.8	3.1	2.4	.5	3.0	---	---
	γ'	14.0	13.4	3.4	1.2	.7	.6	8.6	10.7	---	---
Mar M 200	γ	3.2	.5	20.4	29.4	---	---	0	0	4.2	2.1
	γ'	14.8	13.5	3.1	3.4	---	---	3.7	3.6	4.0	4.9
Nicrotung	γ	.9	8.4	26.1	18.3	---	---	1.0	1.8	2.9	2.1
	γ'	14.9	10.6	3.3	4.9	---	---	7.6	8.2	2.3	3.0
Nimonic 115	γ	4.6	8.8	26.5	19.1	2.9	2.1	.6	2.6	---	---
	γ'	15.7	12.8	4.1	3.8	.6	1.4	7.2	8.4	---	---
Rene 41	γ	1.3	.5	26.8	27.6	7.0	8.2	.7	1.1	---	---
	γ'	9.2	9.3	3.5	5.3	1.3	1.0	10.9	9.2	---	---
TRW 1900	γ	7.6	7.4	24.1	27.3	---	---	.4	.9	3.0	1.8
	γ'	17.4	17.5	3.9	3.9	---	---	1.4	.9	2.6	6.6
Udimet 500	γ	2.3	3.7	28.6	26.7	3.0	2.6	.6	1.0	---	---
	γ'	13.5	10.7	2.9	5.6	1.0	2.1	7.9	8.3	---	---
Udimet 700	γ	5.3	6.8	24.3	24.0	3.9	3.8	1.5	1.3	---	---
	γ'	13.9	12.5	2.7	4.0	.9	1.5	8.1	7.6	---	---
Unitemp AF 1753	γ	2.4	4.4	22.5	18.1	1.1	.9	1.1	2.7	2.7	3.4
	γ'	11.0	7.9	1.3	8.0	.3	.9	11.6	5.9	1.8	0
Waspaloy	γ	1.1	.5	25.0	24.6	3.2	2.6	.7	1.2	---	---
	γ'	9.5	11.2	2.4	4.3	.7	2.1	12.5	10.6	---	---
$\bar{d}$[c]	γ	-0.82		0.99		0.15		-0.69		0.85	
	γ'	.83		-1.5		-.60		.55		-.95	
σ_d	γ	3.1		4.8		.56		.75		1.2	
	γ'	3.1		2.1		.49		1.7		2.4	

[a] Obs is the experimentally observed value (ref. 19).

[b] Est is the value estimated by the calculation.

[c] $$\bar{d} = \sum_{i=1}^{n} \left(\frac{\text{observed - calculated}}{n} \right).$$

TABLE XII. - PHASE OCCURRENCE IN COMMERCIAL ALLOYS

Alloy	Parameters			Phase[b]
	Alloy Ni + Co + Fe,[a] at. %	Gamma Cr + 1.75 × (Mo + W),[a] at. %	Al DN[a]	
Inconel X-750	78.9	17.9	-0.40	n
Mar M 200	73.1	27.4	-.67	n
Inconel 700	72.6	23.6	-.64	n
B-1900	72.5	27.0	-.79	n
Nicrotung	70.7	31.2	-.61	n
Waspaloy	70.5	30.6	-.37	n
GMR 235	69.9	26.2	-.75	--
TRW 1900	69.1	29.4	-.49	n
IN 100	68.7	29.4	-.45	σ
Unitemp AF 1753	68.4	29.2	-.43	n
Nimonic 115	67.7	31.6	-.50	σ
Udimet 500	67.6	33.9	-.44	σ
Udimet 700	67.5	31.1	-.40	σ
Inconel 713C	67.5	31.1	-.53	σ
Rene 41	65.6	39.1	-.34	μ

[a] From Kriege and Baris (ref. 19).

[b] From Collins (ref. 15).

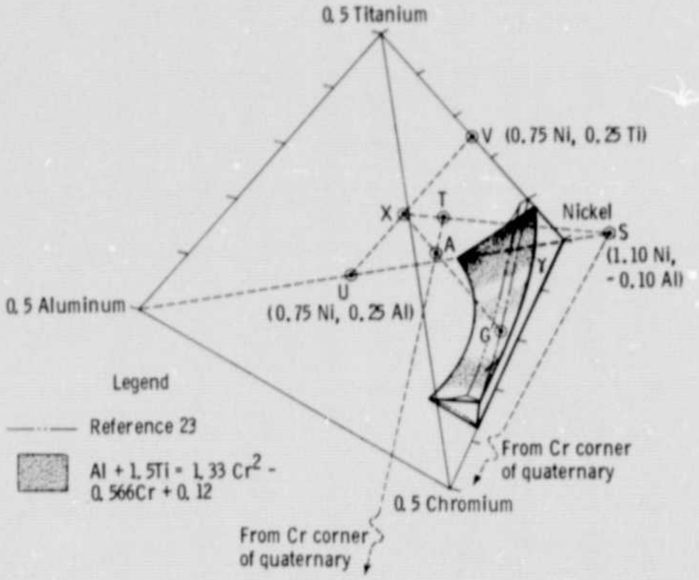


Figure 1. - Nickel-rich region of Ni-Al-Cr-Ti system at 750° C.

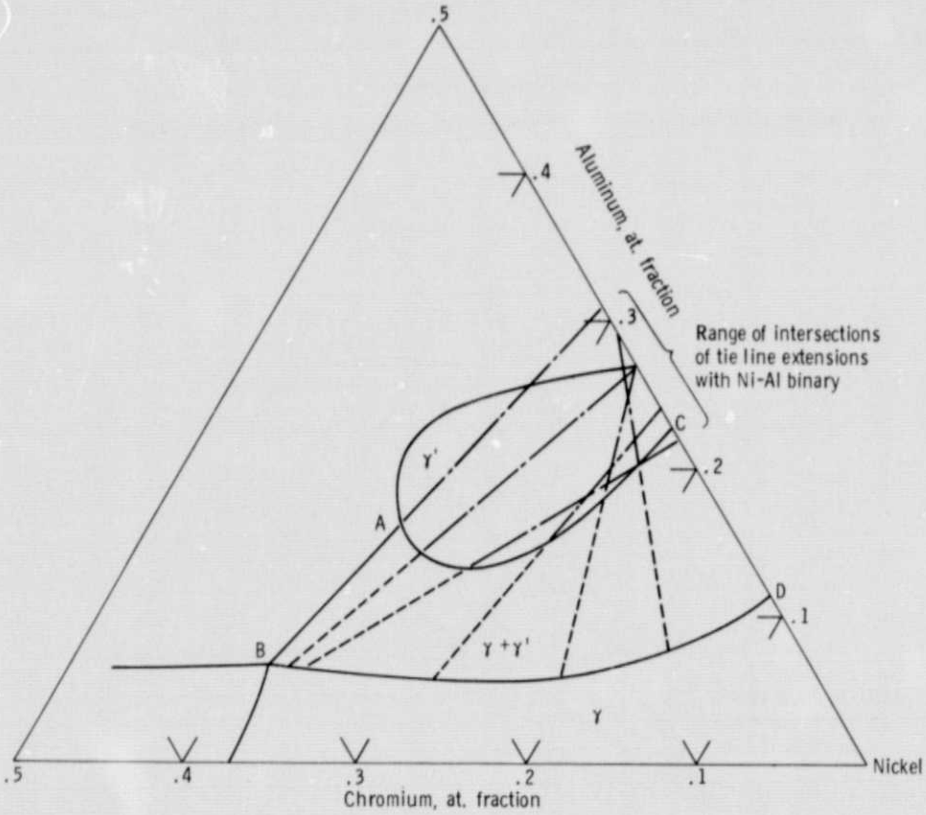


Figure 2. - Nickel-rich region of Ni-Cr-Al system at 750° C (after Taylor and Floyd (ref. 20)).

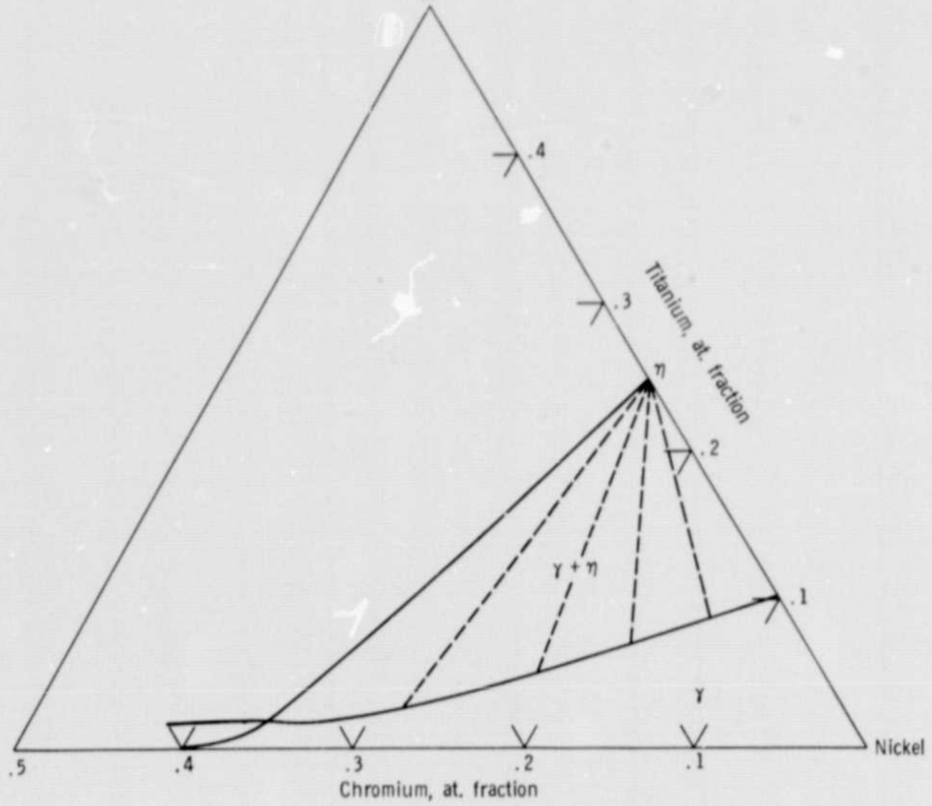


Figure 3. - Nickel-rich region of Ni-Cr-Ti system at 750° C (after Taylor and Floyd (ref. 21)).

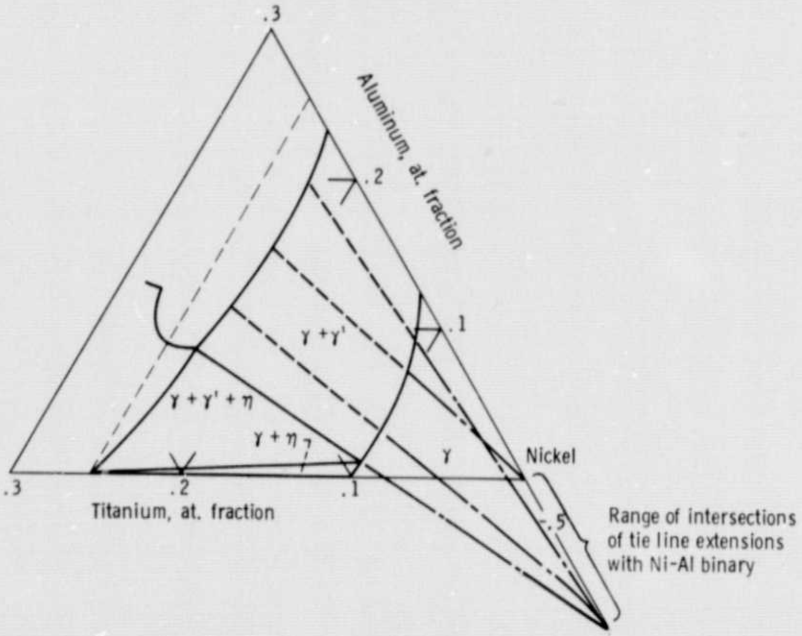
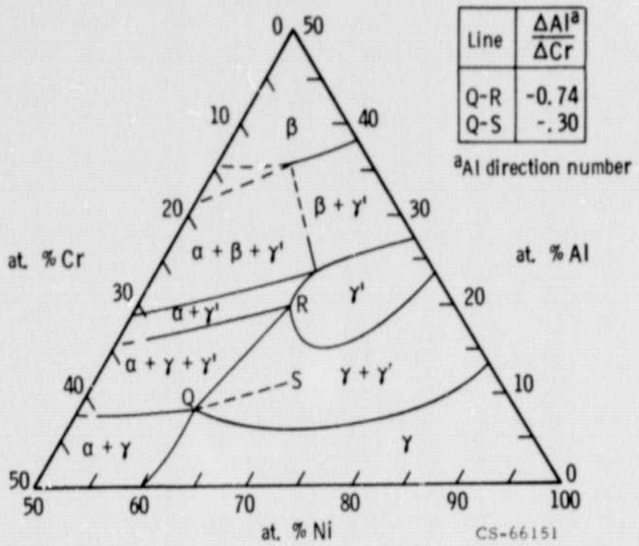


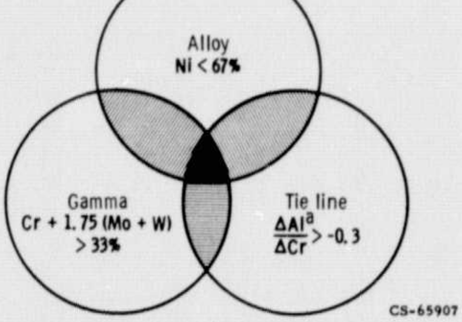
Figure 4. - Nickel-rich region of Ni-Ti-Al system at 750° C (after Taylor and Floyd (ref. 22)).



Line	$\frac{\Delta Al^a}{\Delta Cr}$
Q-R	-0.74
Q-S	-.30

^{a}Al direction number

Figure 5. - The Ni-Cr-Al phase diagram at 850° C from reference 20.



^{a}Al direction number

Figure 6. - Occurrence of BCC and TCP phases in experimental alloys.